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## Memorandum

То	Kristen Durocher	Page 1
CC	Bob Shoemaker, Deb Simmons	
Subject	Summary of Analytical Perspectives HVS Laboratory	Study Results
From	Robert Kennedy	
Date	05/04/2012	

This memorandum provides a summary of the recent laboratory study performed by Gravity Environmental Consulting, LLC (Gravity) and Analytical Perspectives, LLC (AP) in support of the high volume (HV) task component of the Chemical Water Column Monitoring (CWCM) program within the Lower Passaic River Restoration Project (LPRRP) Remedial Investigation and Feasibility Study (RI/FS). This method development work was done by AP and Gravity based on conversations with AECOM representing the Cooperating Parties Group (CPG). AP and Gravity conducted the work independently and not under contract to the CPG, and have shared these results with AECOM to inform the HV sampling program. A summary of the study results written by AP is provided in Appendix A (for polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans [PCDDs/PCDFs]) and Appendix B (for polychlorinated biphenyls [PCBs]) of this document.

The HV program development includes lab experiments to test the Gravity PR-2900 system for sample collection combined with the special analytical protocols designed by AP for the analysis of PCDDs/PCDFs and PCBs. The purpose of these tests was to verify that, in a controlled laboratory environment using large volumes of clean water, the combined sampling-analytical system could produce acceptable recoveries for dynamic spikes (DS), static spikes (SS), and other normal isotope dilution method spikes required by the Environmental Protection Agency (EPA) analytical reference methods. An additional purpose of the study was to compare the performance of two different sorbents, XAD resin and polyurethane foam (PUF), as well as two different media used to deliver the DS, methanol and colloidal silica.

DS were used to measure the capture efficiency of the solid-phase sorbents for the target analytes in the dissolved phase. These spikes are not part of the reference methods, which do not include conditions for high volume sampling. DS using methanol as the delivery medium (Run 1 to Run 4) were introduced slowly into the stream of water immediately before the sorbent cartridge as shown on page 2 of Appendix A. The flow rate for the water stream was 1.5 L/minute and a total of 50 L of water were passed through the vortex and sorbent systems. This DS included the native target analytes (Ax) (includes all 2,3,7,8-PCDD and PCDF isomers in EPA Method 1613B, plus all 209 PCBs listed in EPA Method 1668C) as well a field spike (FS) of four isotope-labeled non-2,3,7,8 isomers, as shown on page 8 of Appendix A, and five isotope-labeled PCBs, as shown on page.6 of Appendix B. This FS set contains compounds intended to represent the range of chemical

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behavior within the full target analyte list and will be used during field sampling as surrogates to monitor capture efficiency. The Ax spike was included in the DS only in the laboratory study to demonstrate comparable Ax and FS performance.

The overall methanolic DS average recovery (i.e., average of XAD and PUF) of all the PCDD/PCDF native target analytes was 85% and the FS average recovery was 83% (see page 3 in Appendix A). This confirms that the FS surrogates behave similar to the native Ax targets and that overall capture efficiency of the sorbents is very good. When the native PCDD/PCDF recoveries in the two different sorbents are compared the PUF appears to have overall better capture efficiency (91%) versus the XAD (78%). The relative recoveries of the native PCBs were not usable due to unexpected contamination by background PCBs in the sampling equipment (as manifested by recoveries greater than 100% and by declining background in each subsequent test). The most likely source of this elevated background is residual contamination from previous uses of the PR-2900. The declining background in each test supports this theory. Associated laboratory method blanks were clean, confirming the analytical finish was not a PCB contaminant source. The labeled PCB FS compounds, however, were not in the 'background contaminant' mix and the mean recovery was 90% for the PUF compared to 79% for the XAD. These slight advantages in the PUF recoveries may not be statistically significant, given the uncertainties in measurement, but it is consistent between the PCDD/PCDF and PCBs and consistent with previous experiments by Gravity using both sorbent media. AECOM, therefore, recommends the PUF sorbent be used for the field sampling in the Passaic River. AECOM also notes that careful efforts to decontaminate the collection device prior to use as well as perform a test of the equipment blank will be critical to the program.

AP also tested a colloidal DS as a potential alternative to the methanolic DS. Colloidal silica of 0.1 um nominal diameter was chosen as an inert carrier for the dynamic spike and it was introduced before the vortex separator and flat filter as shown on page 3 of Appendix A. The results of the colloidal silica DS (Run 5 to Run 8) were not definitive. An average of 33% of the PCDD/PCDF spike and 20% of the PCB FS spike was trapped by the filter in spite of the 0.7 um nominal pore size. On average, only 14% of the PCDD/PCDF and 28% of the PCB FS were recovered from the sorbent media, and roughly 53% of the PCDD/PCDF spike as well as 42% of the PCB FS spike on the colloids was not recovered at all (i.e., it did not appear in the sorbent media or the solids segregated from the water). It appears that a substantial portion of the DS associated with colloidal materials is passing through the PUF and XAD sorbents without extraction, therefore the colloidal DS will not be used in the HV sampling field tests.

SS were added to the sorbents before testing to evaluate potential stripping from the sorbent as water passes through. The five PCDD/PCDF isotope-labeled compounds in the SS exhibited the same average recovery of 91% in both the PUF and XAD sorbents when methanol was used as the spiking medium. The average recovery for the PCB methanolic SS was 94% in both the XAD and PUF runs. Results for the colloidal SS were slightly higher, indicating that if the colloid-associated SS is allowed to associate with the sorbent, subsequent losses from the sorbent are minimal. The difference between the colloidal DS and SS suggests that the colloidal DS does not have sufficient residence/extraction time within the sorbent media under dynamic conditions. All SS recoveries were excellent overall. Details are provided on p.9 of Appendix A and p.13 of Appendix B.

The extraction standards (ES) added to the sorbents after collection but before extraction exhibited an average recovery of 80% for PCDD/PCDF and 84% for the PCBs. ES were added per Methods 1613B and 1668C. Alternate cleanup standards (AS) added to the extract after extraction but before the method required extract cleanups exhibited an average recovery of 73% for the

PCDD/PCDF and 98% for the PCBs. The performance of these ES and AS were in general normal and within method requirements for solid matrices. Details are provided on pages 14 and 18 of Appendix A, and pages 10 and 13 of Appendix B. An anomaly was noted in some ES recoveries in Runs 2, 3, 6, and 8 for the tetra PCDD/PCDFs. These selective losses are thought to be due to isolated problems during cleanup and are not related to the HV sampling method design. The unusually high recoveries for labeled octachlorinated dibenzo-p-dioxin (OCDD) in Run 1 and Run 2 may be related to interferences in the sampling equipment, like the native PCB contaminants, and caused the abnormally low recovery of native OCDD in Run 1 and 2. Other than these anomalies, the ES and AS spike recoveries were acceptable.

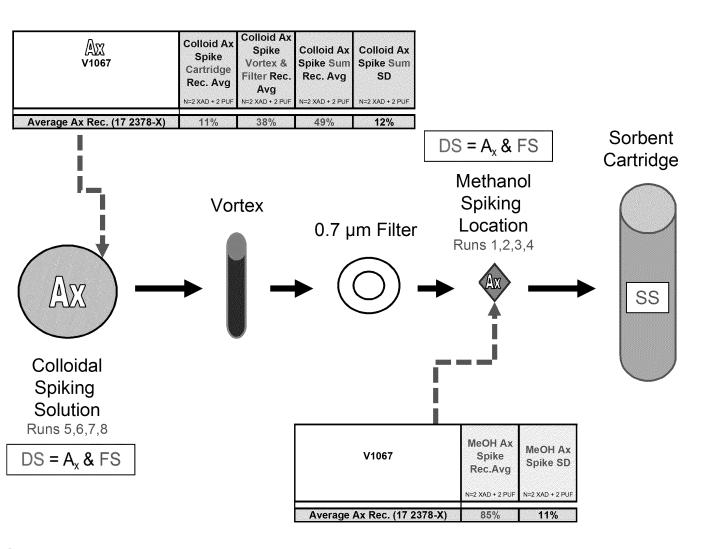
In summary, AECOM concludes the following from the laboratory test:

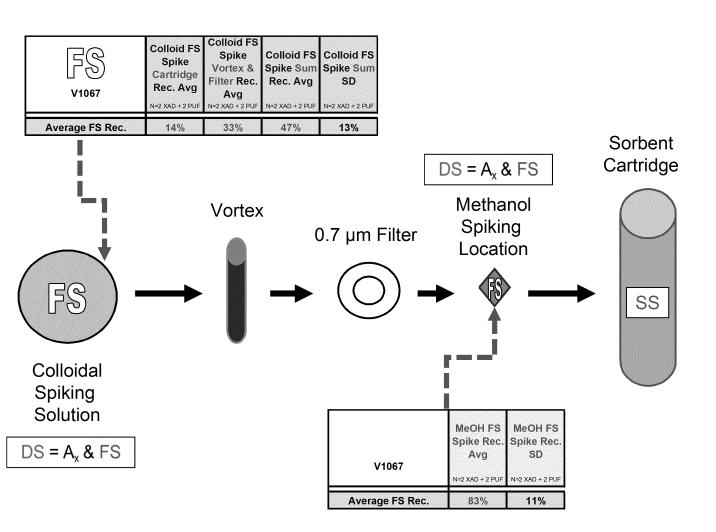
	The combined HV demonstrated very good performance for methanolic DS and SS. Capture efficiency was good and AECOM recommends the methanol spike be used as the FS in the field HV effort and that the FS be introduced immediately before the sorbent cartridge as in the lab study.
	Combined recoveries based on separated solids and solid-phase extraction of the colloidal
	DS were poor, and when compared to the recovery of the colloidal SS, suggest that the
	materials associated with colloids are partially captured as solids but do not have sufficient opportunity to equilibrate with the sorbent media under dynamic conditions. This conclusion
	is independent of the sorbent media type and is very likely to affect any alternative
	sampling protocols that rely on solid-phase extraction as well as separation of bulk solids.
Ш	The methanolic DS recoveries from the PUF media exceeded the recoveries from XAD
	resin on average for both the PCDDs/PCDFs and PCBs. This supports the use of the PUF
	as a more efficient and lower cost approach.
Ш	Performance of ES was in general within acceptable limits per the reference methods.
Ш	Additional cleaning of the Gravity PR-2900 sampling equipment to remove trace level
	PCBs will be required before the field use. The system will be decontaminated per LPRRP
	standard operating procedures in the field, and an equipment blank will be collected to test
	the efficiency of the decontamination procedure.

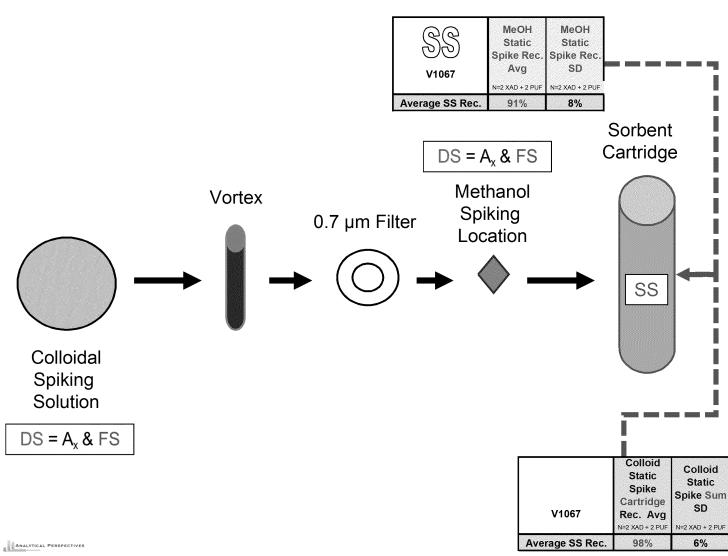
Analytical Perspectives & Gravity Environmental

V1067 HVS 2012 R&D

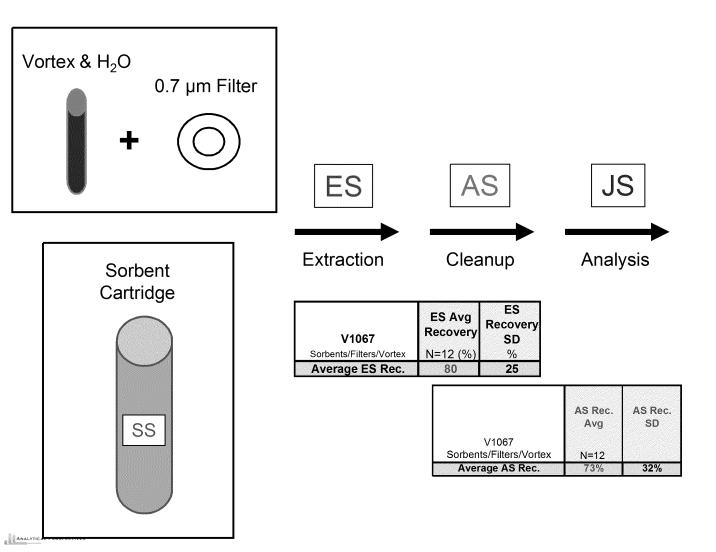
PCDD / Fs

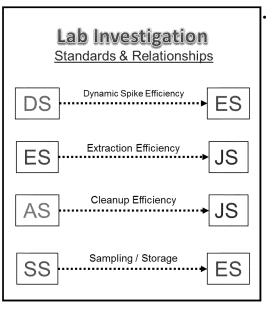






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 $DS = A_x \& FS$ 

PCDD/Fs (native)

Spiked @ 5 pg / 50 L (0.1 ppq)

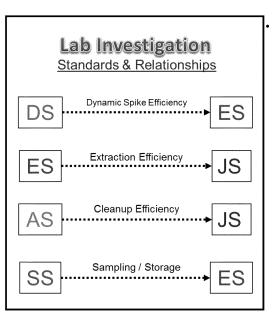
#### **Dynamic Spikes**

- Added during sampling post-filtration and pre-sorbent
- Used to monitor the capture efficiency of the sorbent for <u>both</u> the native target analytes and the labeled analogs used as "surrogates"
- Measured against the extraction standards
- Not part of the EPA reference methods, but based on method principles

#### **Native Recoveries**

Analyte	MeOH Ax Spike Rec.Avg	MeOH Ax Spike SD	Colloid Ax Spike Cartridge Rec. Avg	Spike Vortex & Filter Rec. Avg	Colloid Ax Spike Sum Rec. Avg	Spike Sum SD
	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
2,3,7,8-TCDD	83%	12%	26%	54%	80%	36%
1,2,3,7,8-PeCDD	88%	12%	15%	31%	46%	7%
1,2,3,4,7,8-HxCDD	90%	11%	7%	34%	41%	8%
1,2,3,6,7,8-HxCDD	90%	10%	7%	37%	44%	11%
1,2,3,7,8,9-HxCDD	86%	11%	12%	39%	51%	10%
1,2,3,4,6,7,8-HpCDD	91%	7%	7%	40%	47%	13%
OCDD	51%	31%	6%	34%	40%	14%
2,3,7,8-TCDF	82%	12%	31%	51%	82%	29%
1,2,3,7,8-PeCDF	85%	9%	13%	35%	48%	9%
2,3,4,7,8-PeCDF	85%	7%	17%	27%	45%	6%
1,2,3,4,7,8-HxCDF	90%	11%	6%	38%	44%	11%
1,2,3,6,7,8-HxCDF	87%	9%	6%	39%	45%	8%
2,3,4,6,7,8-HxCDF	87%	8%	9%	37%	46%	6%
1,2,3,7,8,9-HxCDF	83%	11%	7%	40%	47%	8%
1,2,3,4,6,7,8-HpCDF	89%	8%	5%	37%	41%	13%
1,2,3,4,7,8,9-HpCDF	86%	9%	6%	37%	43%	12%
OCDF	84%	12%	6%	32%	38%	8%

details are provided starting at slide #12



#### **Dynamic Spikes**

- Added during sampling post-filtration and pre-sorbent
- Used to monitor the capture efficiency of the sorbent for <u>both</u> the native target analytes and the labeled analogs used as "surrogates"
- Measured against the extraction standards
- Not part of the EPA reference methods, but based on method principles

 $DS = A_x \& FS$ 

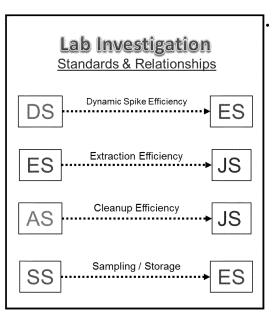
PCDD/FS (native)
Spiked @ 5 pg / 50 L (0.1 ppq)

#### **Native Recoveries**

V1067	MeOH Ax Spike Rec.Avg	MeOH Ax Spike SD	Colloid Ax Spike Cartridge Rec. Avg	Spike Vortex &	Colloid Ax	Spike Sum
	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
Average Ax Rec. (17 2378-X)	85%	11%	11%	38%	49%	12%

details are provided starting at slide #12

ANALYTICAL PERSPECTIVES



#### **Dynamic Spikes**

- Added during sampling post-filtration and pre-sorbent
- Used to monitor the capture efficiency of the sorbent for <u>both</u> the native target analytes and the labeled analogs used as "surrogates"
- Measured against the extraction standards
- Not part of the EPA reference methods, but based on method principles

 $DS = A_x \& FS$ 

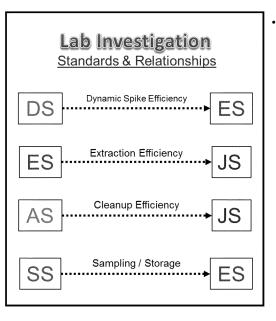
## PCDD/Fs

## Field Spike Standard Recoveries

Analyte	Avg	MeOH FS Spike Rec. SD	Rec. Avg	Colloid FS Spike Vortex & Filter Rec. Avg N=2 XAD + 2 PUF	Colloid FS Spike Sum	Colloid FS Spike Sum SD
FS <sup>13</sup> C <sub>12</sub> -1278-TCDD	87%	7%	31%	23%	54%	12%
FS <sup>13</sup> C <sub>12</sub> -12478-PeCDD	72%	17%	16%	28%	45%	15%
FS <sup>13</sup> C <sub>12</sub> -123468-HxCDD	91%	8%	7%	42%	48%	13%
FS <sup>13</sup> C <sub>12</sub> -1234679-HpCDD	82%	10%	4%	39%	43%	13%

details are provided starting at slide #12

ANALYTICAL PERSPECTIVES



SS

PCDD/Fs

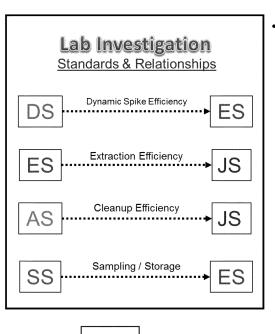
#### Static Spikes

- Added to XAD/PUF in the lab before sampling
- Used to monitor retention of target analytes on the sorbents
- Measured against the extraction standards like target analytes
- Not part of the EPA reference methods, but based on method principles

## Static Spike Standard Recoveries

Analyte	MeOH Static Spike Rec. Avg	MeOH Static Spike Rec. SD	Colloid Static Spike Cartridge Rec. Avg	Colloid Static Spike Sum SD
SS <sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	94%	5%	98%	5%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,7-PeCDD	85%	17%	98%	6%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6-PeCDF	88%	4%	91%	5%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6,9-HxCDF	92%	9%	103%	6%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6,8,9-HpCDF	98%	4%	98%	7%

details are provided starting at slide #12



## PCDD/Fs

ANALYTICAL PERSPECTIVES

Note:

Four of the 12 samples displayed "selective losses"; i.e., TCDD/F recoveries are lower than normal and relative to other congeners. Such losses do not affect TEQ determinations; only congener profiles.

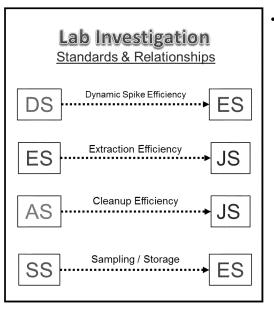
#### Extraction Standards

 Used to measure the native target analytes and static spike in the sorbent by isotope dilution

## **Extraction Standard Recoveries**

Analyte	ES Avg Recovery N=12 (%)	ES Recovery SD %
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	68	30.8
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	78	19.5
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	78	11.1
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	78	9.85
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	78	8.53
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	78	9.16
<sup>13</sup> C <sub>12</sub> -OCDD	145	218
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	64	30.7
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	74	14.4
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	75	20.2
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	78	8.62
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	80	8.41
<sup>13</sup> C <sub>12</sub> -2,3,4,6,7,8-HxCDF	78	7.69
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDF	77	7.77
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	77	7.50
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	80	8.87
<sup>13</sup> C <sub>12</sub> -OCDF	74	8.48

details are provided starting at slide #12





### Alternative Cleanup Standards

Used to monitor the efficiency of cleanups

## Alternate Clean-Up Standard Recoveries

Analyte	AS Rec. Avg N=12	AS Rec. SD
AS <sup>13</sup> C <sub>12</sub> -1368-TCDD	70%	49%
AS <sup>13</sup> C <sub>12</sub> -1368-TCDF	76%	39%

details are provided starting at slide #12

#### Note:

Four of the 12 samples displayed "selective losses"; i.e., TCDD/F recoveries are lower than normal and relative to other congeners. Such losses do not affect TEQ determinations; only congener profiles.



Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	MeOH Ax Spike Rec.Avg	MeOH Ax Spike SD
	pg	pg	pg	pg	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
2,3,7,8-TCDD	91%	93%	67%	83%	83%	12%
1,2,3,7,8-PeCDD	82%	84%	79%	106%	88%	12%
1,2,3,4,7,8-HxCDD	87%	95%	76%	101%	90%	11%
1,2,3,6,7,8-HxCDD	86%	92%	80%	103%	90%	10%
1,2,3,7,8,9-HxCDD	84%	89%	72%	99%	86%	11%
1,2,3,4,6,7,8-HpCDD	88%	91%	84%	101%	91%	7%
OCDD	12%	43%	71%	80%	51%	31%
2,3,7,8-TCDF	88%	92%	64%	82%	82%	12%
1,2,3,7,8-PeCDF	87%	85%	73%	95%	85%	9%
2,3,4,7,8-PeCDF	84%	83%	78%	95%	85%	7%
1,2,3,4,7,8-HxCDF	85%	98%	76%	99%	90%	11%
1,2,3,6,7,8-HxCDF	78%	96%	82%	92%	87%	9%
2,3,4,6,7,8-HxCDF	80%	90%	80%	97%	87%	8%
1,2,3,7,8,9-HxCDF	82%	76%	76%	99%	83%	11%
1,2,3,4,6,7,8-HpCDF	81%	86%	91%	98%	89%	8%
1,2,3,4,7,8,9-HpCDF	81%	97%	77%	88%	86%	9%
OCDF	85%	92%	68%	93%	84%	12%

Analyte	Cartridge Voi	Run 5 Vortex & Filter	Run 5 Combined	l Cartridge I	artridge Vortex &	Run 6 Combined	Run 7 Cartridge PUF 8	Run 7 Vortex & Filter	Run 7 Combined	Run 8 Cartridge XAD 3	Run 8 Vortex & Filter
	pg	pg		pg	pg		pg				pg
2,3,7,8-TCDD	47%	33%	80%	9%	59%	68%	25%	19%	44%	26%	103%
1,2,3,7,8-PeCDD	26%	30%	56%	9%	31%	41%	11%	35%	46%	13%	29%
1,2,3,4,7,8-HxCDD	10%	44%	53%	5%	31%	36%	4%	34%	38%	7%	29%
1,2,3,6,7,8-HxCDD	8%	53%	61%	6%	32%	38%	5%	33%	38%	8%	30%
1,2,3,7,8,9-HxCDD	13%	47%	60%	8%	28%	37%	14%	39%	53%	13%	41%
1,2,3,4,6,7,8-HpCDD	5%	60%	65%	6%	31%	38%	13%	37%	49%	5%	32%
OCDD	5%	56%	61%	5%	25%	29%	9%	27%	36%	5%	29%
2,3,7,8-TCDF	48%	21%	70%	16%	58%	74%	31%	28%	59%	26%	98%
1,2,3,7,8-PeCDF	23%	38%	61%	11%	34%	44%	9%	33%	42%	8%	36%
2,3,4,7,8-PeCDF	29%	24%	53%	14%	29%	43%	15%	30%	45%	12%	26%
1,2,3,4,7,8-HxCDF	6%	53%	59%	7%	35%	42%	6%	35%	41%	6%	29%
1,2,3,6,7,8-HxCDF	5%	52%	56%	5%	34%	39%	10%	36%	45%	3%	35%
2,3,4,6,7,8-HxCDF	14%	41%	55%	5%	38%	43%	8%	34%	42%	7%	36%
1,2,3,7,8,9-HxCDF	5%	52%	58%	6%	33%	39%	8%	37%	46%	8%	39%
1,2,3,4,6,7,8-HpCDF	3%	55%	59%	4%	23%	28%	6%	33%	39%	5%	35%
1,2,3,4,7,8,9-HpCDF	4%	55%	60%	5%	28%	33%	11%	33%	43%	4%	33%
OCDF	4%	43%	47%	5%	22%	27%	9%	31%	40%	5%	31%



Analyte	Colloid Ax Spike Cartridge Rec. Avg	Colloid Ax Spike Vortex & Filter Rec. Avg	Colloid Ax Spike Surr Rec. Avg	Colloid Ax Spike Sum SD
2.3.7.8-TCDD	26%	54%	80%	36%
1,2,3,7,8-PeCDD	15%	31%	46%	7%
1,2,3,4,7,8-HxCDD	7%	34%	41%	8%
1,2,3,6,7,8-HxCDD	7%	37%	44%	11%
1.2.3.7.8.9-HxCDD	12%	39%	51%	10%
1,2,3,4,6,7,8-HpCDD	7%	40%	47%	13%
OCDD	6%	34%	40%	14%
2,3,7,8-TCDF	31%	51%	82%	29%
1,2,3,7,8-PeCDF	13%	35%	48%	9%
2,3,4,7,8-PeCDF	17%	27%	45%	6%
1,2,3,4,7,8-HxCDF	6%	38%	44%	11%
1,2,3,6,7,8-HxCDF	6%	39%	45%	8%
2,3,4,6,7,8-HxCDF	9%	37%	46%	6%
1,2,3,7,8,9-HxCDF	7%	40%	47%	8%
1,2,3,4,6,7,8-HpCDF	5%	37%	41%	13%
1,2,3,4,7,8,9-HpCDF	6%	37%	43%	12%
OCDE	6%	32%	38%	8%

Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	Run 5 Cartridge XAD 4	Run 5 Vortex & Filter	Run 6 Cartridge PUF 7	Run 6 Vortex & Filter %	Run 7 Cartridge PUF 8	Run 7 Vortex & Filter	Run 8 Cartridge XAD 3 %	Run 8 Vortex & Filter
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	97.5	31.2	21.5	72.8	82.7	102.0	88.5	38.6	74.0	96.7	88.5	22.5
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	90.0	60.8	45.9	69.2	92.5	97.9	101.0	74.1	71.4	91.4	91.7	45.2
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	85.5	71.9	64.7	66.7	92.8	88.0	93.3	74.6	67.1	82.5	81.1	62.3
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	85.1	74.2	69.6	65.2	92.8	83.1	94.8	74.0	67.8	82.8	80.8	68.5
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	82.9	76.4	81.3	64.4	90.3	83.8	88.0	71.3	63.2	80.4	76.9	83.0
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	94.7	75.3	79.0	63.0	85.9	82.7	86.6	71.3	64.3	81.8	76.5	80.6
<sup>13</sup> C <sub>12</sub> -OCDD	832.0	157.0	88.6	73.1	82.4	81.8	75.6	63.5	62.3	78.0	74.9	74.0
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	97.7	28.6	20.0	74.1	73.7	101.0	73.9	32.5	74.6	91.8	86.0	18.7
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	83.4	65.5	46.9	65.6	88.1	85.7	91.4	71.9	69.2	84.8	81.6	51.9
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	92.7	54.8	42.3	70.2	93.7	94.6	96.0	67.3	73.2	87.8	86.0	41.3
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	83.2	73.8	75.6	62.1	88.1	86.5	88.7	72.3	64.7	81.9	77.0	76.3
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	86.0	76.9	79.0	65.0	89.6	83.9	93.0	75.2	65.8	81.6	78.1	81.0
<sup>13</sup> C <sub>12</sub> -2,3,4,6,7,8-HxCDF	81.0	74.8	82.9	64.1	87.1	81.6	88.1	71.4	65.5	80.4	79.0	81.0
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDF	80.7	76.8	80.4	61.8	84.8	84.7	86.5	72.2	64.7	80.3	73.6	77.8
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	83.0	73.2	73.2	63.5	86.0	83.0	86.5	70.6	67.7	78.0	77.2	83.0
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	85.3	75.8	81.6	64.6	91.8	86.0	89.3	74.8	64.2	83.3	81.6	85.5
<sup>13</sup> C <sub>12</sub> -OCDF	84.9	72.2	76.4	58.4	81.6	81.5	78.3	64.5	60.8	78.8	74.0	74.2



#### Note:

Four of the 12 samples displayed "selective losses"; i.e., TCDD/F recoveries are lower than normal and relative to other congeners. Such losses do not affect TEQ determinations; only congener profiles.

Analyte	ES Avg Recovery N=12 (%)	ES Recovery SD %
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDD	68	30.8
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDD	78	19.5
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDD	78	11,1
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDD	78	9.85
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDD	78	8.53
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDD	78	9.16
<sup>13</sup> C <sub>12</sub> -OCDD	145	218
<sup>13</sup> C <sub>12</sub> -2,3,7,8-TCDF	64	30.7
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8-PeCDF	74	14.4
<sup>13</sup> C <sub>12</sub> -2,3,4,7,8-PeCDF	75	20.2
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8-HxCDF	78	8.62
<sup>13</sup> C <sub>12</sub> -1,2,3,6,7,8-HxCDF	80	8.41
<sup>13</sup> C <sub>12</sub> -2,3,4,6,7,8-HxCDF	78	7.69
<sup>13</sup> C <sub>12</sub> -1,2,3,7,8,9-HxCDF	77	7.77
<sup>13</sup> C <sub>12</sub> -1,2,3,4,6,7,8-HpCDF	77	7.50
<sup>13</sup> C <sub>12</sub> -1,2,3,4,7,8,9-HpCDF	80	8.87
<sup>13</sup> C <sub>12</sub> -OCDF	74	8.48



Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	MeOH FS Spike Rec. Avg	MeOH FS Spike Rec. SD
	pg	pg	pg	pg	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
FS <sup>13</sup> C <sub>12</sub> -1278-TCDD	81%	93%	81%	94%	87%	7%
FS <sup>13</sup> C <sub>12</sub> -12478-PeCDD	77%	64%	54%	93%	72%	17%
FS <sup>13</sup> C <sub>12</sub> -123468-HxCDD	80%	96%	96%	93%	91%	8%
FS <sup>13</sup> C <sub>12</sub> -1234679-HpCDD	72%	87%	75%	94%	82%	10%



Analyte	Run 5 Cartridge XAD 4	Run 5 Vortex & Filter	Run 5 Combined	Run 6 Cartridge PUF 7	Run 6 Vortex & Filter	Run 6 Combined	Run 7 Cartridge PUF 8	Run 7 Vortex & Filter	Run 7 Combined	Run 8 Cartridge XAD 3	Run 8 Vortex & Filter	Run 8 Combine
	pa	pg		pg	pg		pg	pq		pg	pg	
FS <sup>13</sup> C <sub>12</sub> -1278-TCDD	52%	21%	72%	24%	25%	49%	24%	23%	47%	24%	23%	47%
FS <sup>13</sup> C <sub>12</sub> -12478-PeCDD	30%	35%	66%	12%	26%	38%	13%	30%	43%	11%	21%	32%
FS <sup>13</sup> C <sub>12</sub> -123468-HxCDD	8%	59%	67%	5%	34%	39%	7%	33%	40%	6%	42%	48%
ES 13C12-1234679-HpCDD	2%	60%	62%	3%	33%	35%	5%	31%	36%	6%	32%	38%

Analyte	Colloid FS Spike Cartridge Rec. Avg	Colloid FS Spike Vortex & Filter Rec. Avg	Colloid FS Spike Sum	Colloid FS Spike Sum SD
	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
FS <sup>13</sup> C <sub>12</sub> -1278-TCDD	31%	23%	54%	12%
FS <sup>13</sup> C <sub>12</sub> -12478-PeCDD	16%	28%	45%	15%
FS <sup>13</sup> C <sub>12</sub> -123468-HxCDD	7%	42%	48%	13%
FS <sup>13</sup> C <sub>12</sub> -1234679-HpCDD	4%	39%	43%	13%



Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	MeOH Static Spike Rec. Avg	MeOH Static Spike Rec. SD
	pg	pg	pg	pg	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
SS <sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	98%	92%	89%	98%	94%	5%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,7-PeCDD	100%	73%	68%	97%	85%	17%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6-PeCDF	90%	82%	88%	90%	88%	4%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6,9-HxCDF	98%	90%	79%	100%	92%	9%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6,8,9-HpCDF	96%	100%	103%	94%	98%	4%

Analyte	Run 5 Cartridge XAD 4	Run 6 Cartridge PUF 7	Run 7 Cartridge PUF 8	Run 8 Cartridge XAD 3	Colloid Static Spike Cartridge Rec. Avg	Colloid Static Spike Sum SD N=2 XAD + 2 PUF
SS <sup>37</sup> Cl <sub>4</sub> -2,3,7,8-TCDD	96%	94%	98%	105%	98%	5%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,7-PeCDD	95%	93%	98%	107%	98%	6%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6-PeCDF	90%	89%	89%	98%	91%	5%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6,9-HxCDF	101%	97%	102%	111%	103%	6%
SS <sup>13</sup> C <sub>12</sub> -1,2,3,4,6,8,9-HpCDF	97%	96%	92%	109%	98%	7%



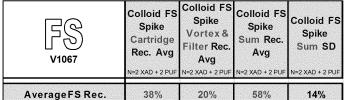
Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5 pg	Run 3 Cartridge XAD 2 pg	Run 4 Cartridge PUF 6 pg	Run 5 Cartridge XAD 4 pg	Run 5 Vortex & Filter pg	Run 6 Cartridge PUF 7 pg	Run 6 Vortex & Filter pg	Run 7 Cartridge PUF 8 pg	Run 7 Vortex & Filter pg	Run 8 Cartridge XAD 3 pg	Run 8 Vortex & Filter pg
AS <sup>13</sup> C <sub>12</sub> -1368-TCDD	99%	30%	20%	96%	68%	98%	72%	38%	98%	103%	99%	18%
AS <sup>13</sup> C <sub>12</sub> -1368-TCDF	99%	40%	29%	97%	85%	99%	87%	52%	96%	98%	98%	27%

Analyte	AS Rec. Avg N=12	AS Rec. SD
AS <sup>13</sup> C <sub>12</sub> -1368-TCDD	70%	34%
AS <sup>13</sup> C <sub>12</sub> -1368-TCDF	76%	29%

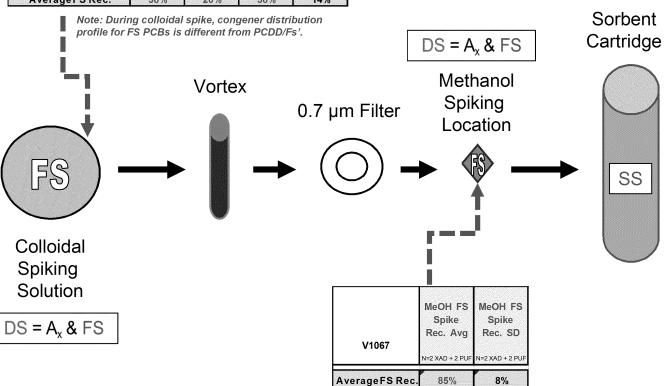
# Analytical Perspectives & Gravity Environmental

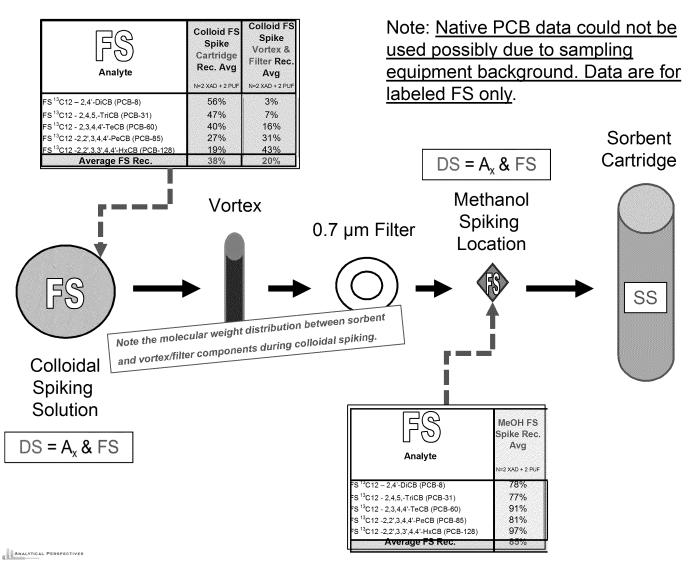
V1067 HVS 2012 R&D

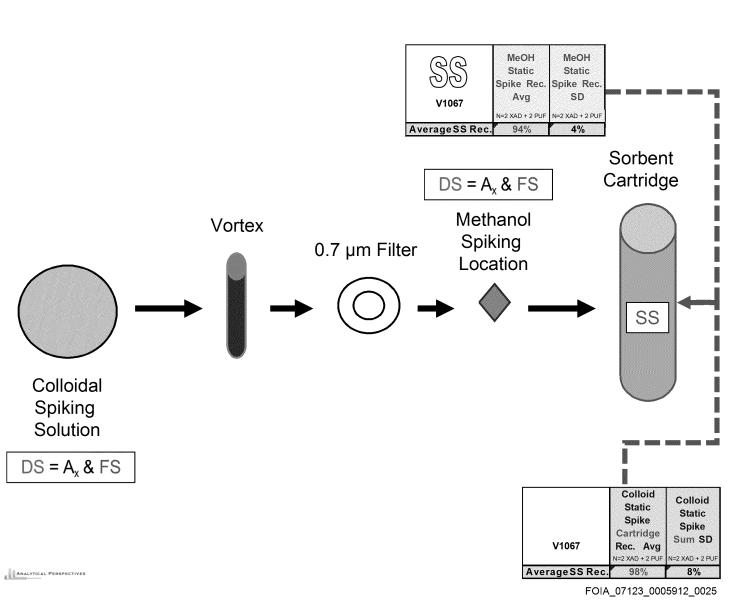
**PCBs** 

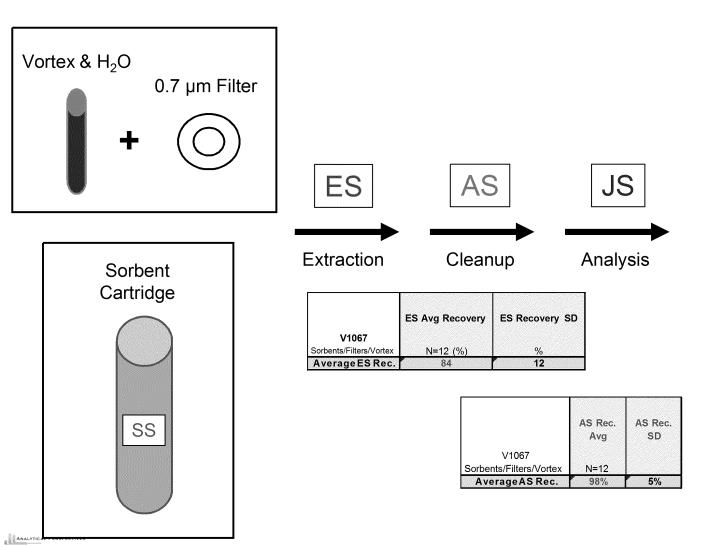


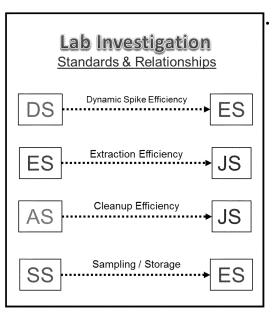
Note: Native PCB data could not be used possibly due to sampling equipment background. Data are for labeled FS only.











#### **Dynamic Spikes**

- Added during sampling post-filtration and pre-sorbent
- Used to monitor the capture efficiency of the sorbent for <u>both</u> the native target analytes and the labeled analogs used as "surrogates"
- Measured against the extraction standards
- Not part of the EPA reference methods, but based on method principles

 $DS = A_x \& FS$ 

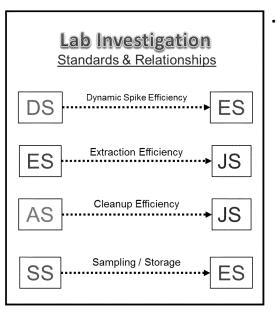
**PCBs** 

Field Spike Standard Recoveries

Analyte	MeOH FS Spike Rec. Avg	MeOH FS Spike Rec. SD	Colloid FS Spike Cartridge Rec. Avg	Colloid FS Spike Vortex & Filter Rec. Avg	IFS Spike	Colloid FS Spike Sum SD
	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
FS <sup>13</sup> C12 – 2,4'-DiCB (PCB-8)	78%	8%	56%	3%	58%	12%
FS <sup>13</sup> C12 - 2,4,5,-TriCB (PCB-31)	77%	9%	47%	7%	54%	15%
FS <sup>13</sup> C12 - 2,3,4,4'-TeCB (PCB-60)	91%	10%	40%	16%	56%	14%
FS <sup>13</sup> C12 -2,2',3,4,4'-PeCB (PCB-85)	81%	5%	27%	31%	58%	13%
FS <sup>13</sup> C12 -2,2',3,3',4,4'-HxCB (PCB-128)	97%	6%	19%	43%	62%	16%

ANALYTICAL PERSPECTIVES

details are provided starting at slide #9



SS

**PCBs** 

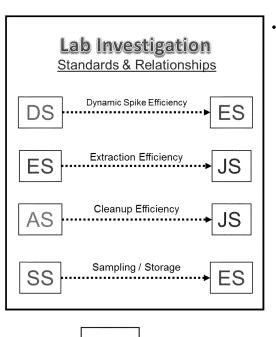
#### Static Spikes

- Added to XAD/PUF in the lab before sampling
- Used to monitor retention of target analytes on the sorbents
- Measured against the extraction standards like target analytes
- Not part of the EPA reference methods, but based on method principles

## Static Spike Standard Recoveries

Analyte	MeOH Static Spike Rec. Avg	Rec. SD	Colloid Static Spike Cartridge Rec. Avg	Colloid Static Spike Sum SD
	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF	N=2 XAD + 2 PUF
SS <sup>13</sup> C <sub>12</sub> -2,4,4'-TrCB (PCB-28)	85%	5%	89%	9%
SS <sup>13</sup> C <sub>12</sub> - 2,3,3',5,5'-PeCB (PCB-111)	97%	5%	102%	7%
SS <sup>13</sup> C <sub>12</sub> - 2,2',3,3',5,5',6-HpCB (PCB-178)	99%	3%	104%	9%

details are provided starting at slide #9



PCBs

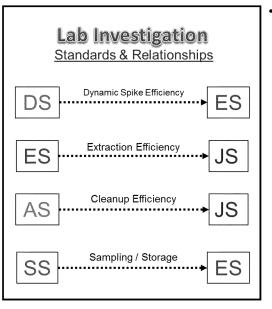
#### Extraction Standards

 Used to measure the native target analytes and static spike in the sorbent by isotope dilution

## **Extraction Standard Recoveries**

Analyte	ES Avg Recovery	ES Recovery SD
7/7/7/7/7	N=12 (%)	N=12 (%)
<sup>13</sup> C <sub>12</sub> - 2-MoCB (PCB-1)	65	13.0
<sup>13</sup> C <sub>12</sub> - 4-MoCB (PCB-3)	72	11.0
<sup>13</sup> C <sub>12</sub> - 2,2'-DiCB (PCB-4)	89	14.2
<sup>13</sup> C <sub>12</sub> - 4,4'-DiCB (PCB-15)	94	11.2
<sup>13</sup> C <sub>12</sub> - 2,2',6-TrCB (PCB-19)	92	11.3
<sup>13</sup> C <sub>12</sub> - 3,4,4'-TrCB (PCB-37)	78	8.7
<sup>13</sup> C <sub>12</sub> - 2,2',6,6'-TeCB (PCB-54)	77	10.2
<sup>13</sup> C <sub>12</sub> - 3,4,4',5-TeCB (PCB-81)	93	10.1
<sup>13</sup> C <sub>12</sub> - 3,3',4,4'-TeCB (PCB-77)	92	10.3
<sup>13</sup> C <sub>12</sub> - 2,2',4,6,6'-PeCB (PCB-104)	77	9.6
<sup>13</sup> C <sub>12</sub> - 2',3,4,4',5-PeCB (PCB-123)	88	7.7
<sup>13</sup> C <sub>12</sub> - 2,3',4,4',5-PeCB (PCB-118)	84	7.9
<sup>13</sup> C <sub>12</sub> - 2,3,4,4',5-PeCB (PCB-114)	89	8.8
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4'-PeCB (PCB-105)	90	8.0
<sup>13</sup> C <sub>12</sub> - 3,3',4,4',5-PeCB (PCB-126)	79	7.2
<sup>13</sup> C <sub>12</sub> - 2,2',4,4',5,5'-HxCB (PCB-153)	88	8.8
<sup>13</sup> C <sub>12</sub> - 2,2',4,4',6,6'-HxCB (PCB-155)	85	12.1
<sup>13</sup> C <sub>12</sub> - 2,3',4,4',5,5'-HxCB (PCB-167)	76	7.8
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4',5-/2,3,3',4,4',5'-HxCB(PCB-156/157)	86	9.8
<sup>13</sup> C <sub>12</sub> - 3,3',4,4',5,5'-HxCB (PCB-169)	<b>6</b> 9	8.2
<sup>13</sup> C <sub>12</sub> - 2,2',3,4',5,6,6'-HpCB (PCB-188)	86	13.9
<sup>13</sup> C <sub>12</sub> - 2,2',3,4,4',5,5'-HpCB (PCB-180)	87	11.3
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,4',5-HpCB (PCB-170)	90	10.4
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4',5,5'-HpCB (PCB-189)	82	8.0
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',5,5',6,6'-OcCB (PCB-202)	88	9.8
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4',5,5',6-OcCB (PCB-205)	76	8.6
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,5,5',6,6'-NoCB (PCB-208)	83	10.6
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,4',5,5',6-NoCB (PCB-206)	94	10.4
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,4',5,5',6,6'-DeCB (PCB-209)	81	11.7

details are provided starting at slide #9





**PCBs** 

## Alternative Cleanup Standards

Used to monitor the efficiency of cleanups

Alternate Clean-Up Standard Recoveries

Analyte	AS Rec. Avg	AS Rec.
AS <sup>13</sup> C12 – 2,4',6-TriCB (PCB-32)	96%	5.6%
AS <sup>13</sup> C12 - 2,2',3',4,5-PeCB (PCB-97)	100%	3.4%
AS <sup>13</sup> C12 -2,2',3,4,5,5'-HxCB (PCB-141)	99%	5.8%

details are provided starting at slide #9



## **ES** Recoveries

Analyte R										Run 8 Cartridge		
	PUF 5	XAD 2	PUF 6	XAD 4	Filter	PUF 7	Filter	PUF 8	Filter	XAD 3	Filter	XAD 1
	%	%	%	%	%	%	%	%	%	%	%	%
<sup>13</sup> C <sub>12</sub> - 2-MoCB (PCB-1)	61.2	54.6	63.6	62.6	87.0	72.4	37.3	62.1	58.4	57.1	72.4	72.0
<sup>13</sup> C <sub>12</sub> - 4-MoCB (PCB-3)	68.6	63.5	67.4	73.8	91.8	79.5	51.0	63.1	66.0	65.3	78.7	78.3
<sup>13</sup> C <sub>12</sub> - 2,2'-DiCB (PCB-4)	83.6	76.6	88.7	89.2	117.0	94.1	62.5	82.2	83.7	82.2	91.6	102.0
<sup>13</sup> C <sub>12</sub> - 4,4'-DiCB (PCB-15)	87.0	89.7	76.5	103.0	110.0	107.0	82.8	79.0	101.0	83.7	102.0	102.0
<sup>13</sup> C <sub>12</sub> - 2,2',6-TrCB(PCB-19)	85.8	79.9	84,7	98,3	109.0	99.7	74.7	79.4	91.0	84.6	91,6	104.0
<sup>13</sup> C <sub>12</sub> - 3,4,4'-TrCB(PCB-37)	69.8	78.8	61.3	83.8	88,6	84.5	67.6	66.6	79.7	79.3	85.4	86.2
<sup>13</sup> C <sub>12</sub> - 2,2',6,6'-TeCB(POB-54)	72.7	68.7	72.2	94.3	85.8	78.3	68.9	61.7	73.7	79.7	70.9	79.8
<sup>13</sup> C <sub>12</sub> - 3,4,4',5-TeOB(POB-81)	83.9	97.2	76.0	105.0	105.0	96.0	84.0	84.1	86.3	97.2	105,0	104.0
<sup>13</sup> C <sub>12</sub> - 3,3',4,4'-TeCB(POB-77)	78.9	89.1	77.3	99.8	107.0	97.1	81.2	85.8	91.2	94.4	104.0	106.0
<sup>13</sup> C <sub>12</sub> - 2,2',4,6,6'-PeCB(PCB-104)	75.2	67.1	68.2	86.5	89.8	86.1	67.1	67.9	79.4	72.6	76.9	70.9
<sup>13</sup> C <sub>12</sub> - 2',3,4,4',5-PeCB (PCB-123)	85.6	87.4	74.8	103.0	94.6	86.8	84.5	77.5	83.5	88.7	91.8	96.6
<sup>13</sup> C <sub>12</sub> - 2,3',4,4',5-PeCB(PCB-118)	80.9	77.5	72.5	99.3	92.6	80.7	79.9	75.7	79.4	84.0	87.1	94.2
<sup>13</sup> C <sub>12</sub> - 2,3,4,4',5-PeCB(PCB-114)	84.2	81.9	76.8	106.0	98.7	87.4	82.7	79.7	82.5	87.7	91.3	98.4
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4'-PeCB (PCB-105)	85.4	84.8	80.3	103.0	102.0	88.7	84.2	79.0	86.2	89.2	92.5	98.7
<sup>13</sup> C <sub>12</sub> - 3,3',4,4',5-PeOB(POB-126)	79.4	81.3	68.5	95.9	80.7	79.0	81.8	67,7	73,1	80.0	83.8	81.0
<sup>13</sup> C <sub>12</sub> - 2,2',4,4',5,5'-HxCB(PCB-153)	83.1	82.0	76.8	97.6	105.0	92.7	78.8	78.8	88.7	86.8	89.5	87.9
<sup>13</sup> C <sub>12</sub> - 2,2',4,4',6,6'-HxCB(PCB-155)	75.8	69.1	68.7	88.3	105.0	96.9	72.1	74.7	93.0	77.0	91.3	89.8
<sup>13</sup> C <sub>12</sub> - 2,3',4,4',5,5'-HxCB(PCB-167)	67.5	72.9	61.2	85.3	87.3	80,0	72.4	65.4	78.9	78.1	76.9	83.6
13C <sub>12</sub> - 2,3,3',4,4',5-/2,3,3',4,4',5'-HxCB(PCB-156/157)	75.7	79.1	67.9	97.9	94,8	91.8	82.5	73.5	87.3	86,1	87.6	102.0
<sup>13</sup> C <sub>12</sub> - 3,3',4,4',5,5'-HxCB(PCB-169)	63.9	69.8	52.9	82.3	75.5	78.0	68.8	56.3	69.4	66.5	75.2	71.0
<sup>13</sup> C <sub>12</sub> - 2,2',3,4',5,6,6'-HpCB(PCB-188)	86.3	85.5	76.1	104.0	85.9	89.0	74.5	71.4	74.5	78.4	79.0	120.0
<sup>13</sup> C <sub>12</sub> - 2,2',3,4,4',5,5'-HpCB(PCB-180)	84.9	85.1	79.8	101.0	92.1	92.0	74.6	71.8	78.0	83.2	82.3	110.0
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,4',5-HpCB(PCB-170)	82.4	77.7	79.5	97.9	107.0	97.5	75,8	82.0	87.7	85.9	90.1	99.8
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4',5,5'-HpCB(PCB-189)	74.8	75.8	67.1	92.5	92.4	89.8	77.4	71.4	82.9	83.5	87.9	84.7
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',5,5',6,6'-OcCB(PCB-202)	82.5	77.2	77.1	101.0	97.3	93.8	78.9	78.8	84.3	85.4	80.0	103.0
<sup>13</sup> C <sub>12</sub> - 2,3,3',4,4',5,5',6-OcCB(PCB-205)	71.1	69.1	62.6	83,4	86.9	81.2	66.8	69.4	72.6	73.8	74.9	86.0
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,5,5',6,6'-NoCB(PCB-208)	78.0	73.8	71.8	91.7	95.7	94.2	69.7	77.0	78.2	76.1	78.5	84.2
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,4',5,5',6-NoCB(PCB-206)	87.2	83.0	80.0	107.0	103.0	102.0	85.3	82.0	87.6	91.2	92.7	107.0
<sup>13</sup> C <sub>12</sub> - 2,2',3,3',4,4',5,5',6,6'-DeCR(PCB-209)	74.7	68.6	68.6	90.8	99,8	90.6	67.6	75.9	79.8	75.1	77.2	83.2





## **FS** Recoveries

Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	MeOH Spike Mean	MeOH Spike SD
FS <sup>13</sup> C12 – 2,4'-DiCB(PCB-8)	77%	79%	67%	87%	78%	8%
FS <sup>13</sup> C12 - 2,4,5,-TriCB (PCB-31)	72%	84%	67%	85%	77%	9%
FS <sup>13</sup> C12 - 2,3,4,4'-TeCB(PCB-60)	84%	96%	81%	103%	91%	10%
FS <sup>13</sup> C12 -2,2',3,4,4'-PeCB (PCB-85)	80%	85%	75%	86%	81%	5%
FS <sup>13</sup> C12 -2,2',3,3',4,4'-HxCB (PCB-128)	88%	97%	101%	102%	97%	6%



## **FS** Recoveries

Analyte	Run 5 Cartridge XAD 4	Run5 Vortex & Filter	Run5 Combined	Run 6 Cartridge PUF 7	Run 6 Vortex & Filter	Run 6 Combined	Run7 Cartridge PUF 8	Run 7 Vortex & Filter	Run 7 Combined	Run8 Cartridge XAD 3	Run8 Vortex & Filter	Run8 Combined
FS <sup>13</sup> C12 – 2,4'-DiCB(PCB-8)	66%	4%	70%	46%	2%	49%	46%	2%	48%	65%	2%	67%
FS <sup>13</sup> C12 - 2,4,5,-TriCB(PCB-31)	67%	7%	74%	41%	8%	49%	33%	7%	40%	48%	6%	54%
FS <sup>13</sup> C12 - 2,3,4,4'-TeCB(PCB-60)	61%	16%	77%	33%	12%	45%	30%	20%	50%	35%	18%	53%
FS <sup>13</sup> C12 -2,2',3,4,4'-PeCB(PCB-85)	46%	30%	77%	22%	32%	54%	18%	29%	47%	23%	31%	54%
FS <sup>13</sup> C12 -2,2',3,3',4,4'-HxCB (PCB-128)	36%	50%	86%	12%	41%	53%	14%	41%	55%	15%	40%	55%

Analyte	Colloid FS Spike Cartridge Rec. Avg	Colloid FS Spike Vortex & Filter Rec. Avg	Colloid FS Spike Sum Rec. Avg	Colloid FS Spike Sum SD
FS <sup>13</sup> C12 – 2,4'-DiCB (PCB-8)	56%	3%	58%	12%
FS <sup>13</sup> C12 - 2,4,5,-TriCB (PCB-31)	47%	7%	54%	15%
FS <sup>13</sup> C12 - 2,3,4,4'-TeCB (PCB-60)	40%	16%	56%	14%
FS <sup>13</sup> C12 -2,2',3,4,4'-PeCB (PCB-85)	27%	31%	58%	13%
FS <sup>13</sup> C12 -2,2',3,3',4,4'-HxCB (PCB-128)	19%	43%	62%	16%



## SS Recoveries

Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	MeOH Static Spike Rec. Avg	MeOH Static Spike Rec. SD
SS <sup>13</sup> C <sub>12</sub> -2,4,4'-TrCB (PCB-28)	88%	89%	77%	86%	85%	5%
SS <sup>13</sup> C <sub>12</sub> - 2,3,3',5,5'-PeCB (PCB-111)	103%	98%	93%	94%	97%	5%
SS <sup>13</sup> C <sub>12</sub> - 2,2',3,3',5,5',6-HpCB (PCB-178)	97%	100%	104%	98%	99%	3%

Analyte	Run 5 Cartridge XAD 4	Run 6 Cartridge PUF 7	Run 7 Cartridge PUF 8	Run 8 Cartridge XAD 3	Colloid Static Spike Cartridge Rec. Avg	Colloid Static Spike Sum SD
SS <sup>13</sup> C <sub>12</sub> -2,4,4'-TrCB (PCB-28)	94%	88%	77%	99%	89%	9%
SS <sup>13</sup> C <sub>12</sub> - 2,3,3',5,5'-PeCB (PCB-111)	98%	102%	97%	111%	102%	7%
SS <sup>13</sup> C <sub>12</sub> - 2,2',3,3',5,5',6-HpCB (PCB-178)	107%	99%	96%	115%	104%	9%



## **AS Recoveries**

Analyte	Run1 Cartridge XAD 1	Run 2 Cartridge PUF 5	Run 3 Cartridge XAD 2	Run 4 Cartridge PUF 6	Run 5 Cartridge XAD 4	Run 5 Vortex & Filter	Run 6 Cartridge PUF 7	Run 6 Vortex & Filter	Run 7 Cartridge PUF 8	Run 7 Vortex& Filter	Run 8 Cartridge XAD 3	Run 8 Vortex & Filter
AS 13C12 – 2,4',6-TriCB (PCB-32)	105%	95%	96%	94%	98%	101%	95%	89%	95%	99%	83%	97%
AS 13C12 - 2,2',3',4,5-PeCB (PCB-97)	98%	99%	99%	101%	102%	107%	97%	100%	100%	96%	100%	106%
AS 13C12 -2,2',3,4,5,5'-HxCB (PCB-141)	96%	98%	101%	95%	102%	109%	100%	90%	100%	94%	92%	106%

Analyte	AS Rec. Avg	AS Rec. SD
AS <sup>13</sup> C12 – 2,4',6-TriCB (PCB-32)	96%	5.6%
AS <sup>13</sup> C12 - 2,2',3',4,5-PeCB(PCB-97)	100%	3.4%
AS <sup>13</sup> C12-2,2',3,4,5,5'-HxCB (PCB-141)	99%	5.8%

